

This Page Is Inserted by IFW Operations  
and is not a part of the Official Record

## **BEST AVAILABLE IMAGES**

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images may include (but are not limited to):

- BLACK BORDERS
- TEXT CUT OFF AT TOP, BOTTOM OR SIDES
- FADED TEXT
- ILLEGIBLE TEXT
- SKEWED/SLANTED IMAGES
- COLORED PHOTOS
- BLACK OR VERY BLACK AND WHITE DARK PHOTOS
- GRAY SCALE DOCUMENTS

**IMAGES ARE BEST AVAILABLE COPY.**

As rescanning documents *will not* correct images,  
please do not report the images to the  
Image Problem Mailbox.



CASE GTW-21923/VAC 533

#8  
0130/03  
PS**CERTIFICATE OF MAILING**

I hereby certify that this paper (along with any paper referred to as being attached or enclosed) is being deposited with the United States Postal Service on the date shown below with sufficient postage as first class mail in an envelope addressed to the Assistant Commissioner for Patents, Washington, D.C. 20231.

\_\_\_\_\_  
Type or print name\_\_\_\_\_  
Signature\_\_\_\_\_  
Date**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

IN RE APPLICATION OF

ANNE FLISHER ET AL

APPLICATION NO: 09/890,129

FILED: JULY 27, 2001

FOR: POLYMERISATION PROCESS

Group Art Unit: 1711

Examiner: S. Berman

**RECEIVED**  
JUN 18 2003  
TC 1700

Assistant Commissioner for Patents

Washington, D.C. 20231

**DECLARATION UNDER RULE 1.131**

The purpose of this Declaration is to establish a date of invention in Great Britain at a date prior to October 6, 1999, which is the effective date of U.S. Patent No. 6,262,141 that was cited by the Examiner.

The persons making this Declaration are the inventors.

To establish a date of invention of this application, the following attached documents are submitted as evidence:

laboratory notebook records as follows:

a) From notebook Number 76 of Anne Flisher pages 38 to 47, 50 to 54, 68 to 69, 73 to 75, 92 to 94, 111 to 113, 145, 148 to 150, 166, 181, 191.

b) From notebook number 91 of Angela Holman working under the supervision of Anne Flisher pages 69 to 70, 96 to 97, 136 to 137, 154 to 155, 160 to 161.

c) From notebook number 97 of Anne Flisher pages 1, 15 to 16, 19 to 21, 33 to 34, 38.

The dates on the above documents have been masked.

All the work recorded in the above notebook pages and recorded in the reports was carried out before 5 October 1999. The project was initially numbered 1542. Subsequently the number was changed to 2006 and the project given the code name "Bonsai".

From these documents, it can be seen that the invention of this application was conceived and reduced to practice at least by the date of October 5, 1999, which is a date earlier than the effective date of the reference.

The features of the claims of record are found in the notebooks in accordance with the following Table;

Claim	Conception/reduction to practice.
	Book Reference: Book number/Page number
1	76/42
2	76/40
3	97/33
4	76/41
5	76/42
6	76/41
7	76/42
8	91/69
9	91/69
10	97/15
11	76/43
17	76/42 and 97/33
18	97/33
19	76/74 and 76/75

This Declaration is being submitted prior to final rejection.

As a person signing below:

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

**Signatures:**

Full name of first joint inventor **Anne Ellisner**

Inventor's signature *anne ellisner* Date 10/06/03  
month/day/year

Residence 5 Warley View  
Highroad Well  
Halifax  
West Yorkshire  
HX2 0AG  
Great Britain

Citizenship British

Post Office Address - same as above

Full name of second joint inventor **Gareth Ian Naylor**

Inventor's signature *Gareth Naylor* Date 10/06/03  
month/day/year

Residence 23 Thornton Street  
Halifax  
West Yorkshire  
HX1 3SD  
Great Britain

Citizenship British

Post Office Address - same as above

09/090,129

- 3 -


GTW-21923/AAC 533

**Residence** 23 Thornton Street  
Halifax  
West Yorkshire  
HX1 3SD  
Great Britain

**Citizenship** British

**Post Office Address** – same as above

**Full name of third joint inventor** Lee Raistrick

**Inventor's signature** 

**Date** 2/5/03  
month/day/year

**Residence** 78 Sefton Avenue  
Hove Edge  
Brighouse  
West Yorkshire  
HD6 2NA  
Great Britain

**Citizenship** British

**Post Office Address** – same as above

## ARCHIVE DETAILS

CONTROL No.

ROLL START

END

IND

Project No. 1542

Project "Iota" Reduction of  
Residual Acrylamide

In order to investigate the possibility of reducing residual acrylamide a number of ~~investigations~~ experiments were to be carried out. Pure raw materials was the first possibility. Solid acrylamide was obtained (Mitsubishi) and was used in the make-up of monomer.

- Magnafloc 351 was chosen as the product due to its high acrylamide content (94%) and its simplicity (cf m303 which contains  $\text{NaO}_4$  which could be a source of error)
- De-ionised water was used in the make up of the monomer.

~~The following table is a~~

The following contained solid acm and DiW

Poly N°	$\text{KBrO}_3$	$\text{Na}_2\text{SO}_3$	AZDN	AcVA
132-2915	3.5	7.0	300	100
2916	3.5	7.0	300	100
2917	5.0	10.0	300	100
2918	5.0	10.0	300	100

Another set of four <sup>monomers</sup> ~~polymers~~ were made and initiated using the same levels. These monomers contained solid acm and tap water. They were

I declare that this work was done by me

*after*

I declare that this work was explained to me and that I have understood it

*Robert*

## ARCHIVE DETAILS

CONTROL No.

ROLL

START

END

IND?

Project No.

FPR Nos 132-2919 → 2922.

- A third set of standard polymers were produced (FPR Nos 132-2923 → 2926) these contained plant produced acrylamide and tap water.

RESULTS

The polymerisation data is as follows:-

FPR No	KBrO <sub>3</sub>	Na <sub>2</sub> S <sub>2</sub> O <sub>8</sub>	T <sub>0</sub>	T <sub>10</sub>	T <sub>20</sub>	T <sub>30</sub>	T <sub>40</sub>	T <sub>60</sub>	T <sub>80</sub>	T <sub>max</sub>	Poly Time	Ind Time	* = 85 mins
132-2915	3.5	7	-1	0	2	5	9	25	75*	82	110	4min	PURE
2916	3.5	7	-1	1	3	5	8	22	66*	84	110	30sec	AcM
2917	5.0	10	-1	4	7	13	25	81	85*	85	85	50sec	+ DIW
2918	5.0	10	-1	1	6	14	28	81	82*	82	85	40sec	
2919	3.5	7	-1	1	2	5	8	20	51	84	110	30sec	PURE
2920	3.5	7	-1	1	4	5	8	19	46	84	110	26sec	AcM
2921	5.0	10	0	2	5	10	16	57	85	85	80	30sec	+ TAP
2922	5.0	10	0	2	FAILED TO POLYMERISE Bubbles out @ 20mins								H <sub>2</sub> O
2923	3.5	7	-1	1	3	6	9	22	51	84	110	20sec	PLANT
2924	3.5	7	0	2	4	6	8	16	34	83	120	20sec	AcM
2925	5.0	10	-1	2	6	13	22	61	82	82	80	10sec	+ TAP
2926	5.0	10	0	4	9	18	31	78	81	81	80	10sec	H <sub>2</sub> O

The resulting polymers were assessed for solubility and viscosity<sup>3</sup> and residual acrylamide :-

I declare that this work was done by me

I declare that this work was explained to me and that I have understood it

## ARCHIVE DETAILS

CONTROL No. ROLL START END IND:

Project No.

FREN°	Viscosity@25°C	Solubility	Res acm %
B2-2915	21	Good	0.088
2916	21	Good	0.099
2917	18	Good	0.085
2918	18	Good	0.089
2919	21	Good	0.108 98
2920	21	Good	0.105 98
2921	17	Good	0.089
2922	Did not polymerise		
2923	24	Fair	0.091
2924	23	Fair	0.093
2925	21	Fair	0.090
2926	19	Fair	0.096

Visc Spec

17-23-29

The above results suggest that the use of the pure acrylamide did not lead to a reduction in residual acrylamide. These results show no change in viscosity when Deionised water was substituted for tap water.

The next stage was to polymerise in the presence of very high thermal initiators. AzDN and AcVA were to be used in 1000ppm quantities. The thermals were to be dissolved in methanol, a well known chain transfer agent. Only one ~~per~~ monomer was initiated to make sure it polymerised. The levels chosen were as follows:-

I declare that this work was done by me

A. J. O'Neil

I declare that this work was explained to me and that I have understood it

R. J. O'Neil



## ARCHIVE DETAILS

CONTROL No. ROLL START END IND7

Project No.

Poly number	KBrO <sub>3</sub>	Na <sub>2</sub> SO <sub>3</sub>	AZDN*	ACVA*
132-2927	3.5	7	1000	1000

~~5%~~  
\* Solutions (5%) were made up and added during the 30 min. degas period. 60mls of each initiator was added. This volume of liquid had the effect of warming the monomer slightly. This may have affected the polymerisation slightly. The polymerisation details are as follows.

Poly No.	Initial Temp °C	T <sub>1.5</sub>	T <sub>2.0</sub>	T <sub>4.0</sub>	T <sub>5.0</sub>	T <sub>6.0</sub>	T <sub>9.0</sub>	Poly Time (mins)	Induc Time (mins)
132-2927	2	4.5	5	11	16	22	81	110	11

Three further monomers were initiated

(132-2928) - One contained normal thermal levels (300 AZDN : 100 ACVA and 120 ml methanol.

(132-2929) - One contained standard thermal and 1000ppm of a ultra violet initiator (2030 mls) BEE (m.c)

(132-2930) - One was a standard with no methanol and ~~the~~ normal thermal initiator levels (300 : 100)

BEE = Benzoin ethyl ether

The polymerisation details are shown overleaf:-

I declare that this work was done by me

*John*

I declare that this work was explained to me and that I have understood it

*John*

ARCHIVE DETAILS

CONTROL No. ROLL START END IND

Project No.

Poly No.	T <sub>0</sub> °C	T <sub>10</sub> °C	T <sub>20</sub> °C	T <sub>40</sub> °C	T <sub>60</sub> °C	T <sub>80</sub> °C	T <sub>120</sub> °C	Poly Time (mins)	Ind Time (mins)
132-2928	2	3	5	7	13	24	79	140	#
132-2929	2	4	5	9	14	24.5	80.5	140	#
132-2930	1	2	4	6	12	20	70	140	#

# The bubblers were removed at 25 mins. ~~and the~~ The last two polys had not polymerised, timing continued.

Peak temperatures were achieved even though the rate was slow. Testing was carried out the results are as follows

No	Visc <sup>1</sup>	Visc <sup>2</sup>	Solubility	Residual Acryl
132-2927	13	16	Good	0.117
132-2928	17	17	Good	0.135
132-2929A	20	19	F/Good	0.102
132-2929B	21	21	F/Good	0.078
132-2930	25	22	F/Good	0.121

Visc Spec

17-23-2-1

1020 ppm

150 ppm w treated

As poly n° 132-2929 contained an ultra violet initiator it was dried conventionally (100g) and 100g was uv treated for 30 mins before FBD drying. It is hoped that uv treatment would convert any residual acm to polymer.

In order to reduce residual monomer it was suggested that reducing the solids content of the monomer, short stopping the polymerisation and including a high temperature acting thermal initiator to ~~take~~ mop up any remaining

I declare that this work was done by me

apover

I declare that this work was explained to me and that I have understood it

RDP

## ARCHIVE DETAILS

CONTROL No. ROLL START END IND?

Project No.

monomer. Hopefully the peak temperature of each polymer will be reduced to approximately <sup>60</sup>°C ~~at which point~~ <sup>this leaves</sup> the remaining thermal ~~should~~ <sup>be</sup> activate during drying process.

The following monomers were prepared

Poly No.	KBrO <sub>3</sub>	Na <sub>2</sub> SO <sub>3</sub>	AZDN	Acua %	solids
132-2931	3.5	7	300	100	30
132-2932	3.5	7	400	—	20
132-2933	3.5	7	—	400	20
132-2934	3.5	7	300	100	20

Polys 132-2932 → 2934 peaked at approx 45 after 4 hours. They were left overnight in the fume cupboard. These polys were not used further. (132-2931 was ok) residual Ultra violet drying is a possibility for the reduction of Acua. A ~~test~~ preliminary experiment was set up to find out the effects of uv light treatment.

- Wet gel was lubricated using 0.1% Bionic solution (100g) - and
- BEE (Benzoyl Ethyl ether) was added (1ml/2ml) of a 10% <sup>soln</sup> in methanol
- 100g was dried traditionally in a FBD.
- 100g was dried in a FBD with uv light shining through a glass head.

The residual monomer had already been assessed on the FBD dried gel.

I declare that this work was done by me

almer

I declare that this work was explained to me and that I have understood it

R. Brown

## ARCHIVE DETAILS

CONTROL No.

ROLL

START

END

IND?

Project No.

Poly N°	Visc	Res Acn <sup>standard</sup>	UV Init
132-2924 A	23	0.093	2g ml → UV treated / dried
132-2924 B	23	"	2ml → not — " —
132-2925 A	21	0.090	1ml → UV treated / dried
132-2925 B	21	0.090	1ml → not — " —

\* 132-2924B and 132-2925B were UV treated / dried.

The glass head with UV light equipment dried the wet gel in approx 30 mins, the traditional method took longer (60 mins-). The above will be assessed for residual acn.

Poly N°	Res. Acn %	Viscosity <sup>1</sup>	Viscosity <sup>2</sup>	Solubility
132-2924 A	0.092	-	-	F/G skd
2924B	0.052	18	19	F/G +UV
2925A	0.092	18	17	F/G sk
2925B	0.032	14	14	F/G +UV
[2931-2931E] 2931A	0.023 MeOH wash	23	23	F
2931B	0.018 MeOH wash	22	23	F
2931E	0.066 microwave	T-T-L	T-T-L	F
2931D	0.048 uv dried, no initiator	19	20	F

T-T-L - Torque too low.

In each case the UV treatment / drying proved effective, reducing residual monomer by up to 2/3. MeOH washing was very useful in reducing res acn but is not a feasible option for use on the plant. Microwave + uv drying was also effective.

I declare that this work was done by me

*of Onen*

I declare that this work was explained to me and that I have understood it

*PPH*

## ARCHIVE DETAILS

CONTROL No.

ROLL

START

END

IND?

--	--	--	--	--

Project No.

Methanol washing/drying:

- Standard M351 product was manufactured (FPR No. 132-2931)
- Wet gel was worked up and minced as usual.
- This gel was then treated with aqueous methanol solutions.

It was hoped that this treatment would extract any unreacted acrylamide. The use of aqueous methanol is important as the presence of water swells the gel exposing more unreacted monomer to the alcohol for extraction.

- A ~50% aq MeOH solution was used in the first instance and increasing MeOH concentrations thereafter until pure MeOH (100%) was used.
  - This lead to a white chip being produced. The treated chip was not completely dry and so was placed in the oven overnight @ 50°C.
  - A second attempt at methanol drying was made using a 70% sol<sup>n</sup> to swell the gel. This mixture was mixed using a hand blender and left for ~15 mins.
  - 95% was then used followed by 100%.
  - This was also dried overnight in the oven. ~~Then~~
- The two products were viscosity tested and tested for residual acrylamide. Although this MeOH washing is not viable on the plant due to the volume of Methanol used, it is still valuable to check if residual monomer is reduced.

I declare that this work was done by me

*of Ther*

I declare that this work was explained to me and that I have understood it

*[Signature]*

Project No.

As a comparison, this product (132-2931) was dried using the microwave and ~~conventionally and under~~ ultra violet. The product was dried for 10mins @ 400Watts and 45mins @ 150Watts. 100g of polymer was dried in each instance.

The results for these drying treatments are shown on page 44.

Four more polymers were produced using works produced acrylamide. The monomers were made up using the standard M361 recipe but contained 25% solids (cf 30% standard). This was in order to lower the peak temp of the polys and allow the thermal initiators to activate ~~while the~~ during oven residence/drying.

The following polymers were produced

Poly. N°	KBrO <sub>3</sub>	Na <sub>2</sub> SO <sub>3</sub>	AzDN	AcVA
132-2935	3.5	7	300	100
2936	3.5	7	400	—
2937	3.5	7	—	400
2938	3.5	7	100	300

25%  
solids.

The polymerisation data was as follows:-

I declare that this work was done by me

*after*

I declare that this work was explained to me and that I have understood it

*[Signature]*

## ARCHIVE DETAILS

CONTROL No. ROLL START END IND?

IND?

Project No.

Poly N°	T <sub>0</sub>	T <sub>10</sub>	T <sub>20</sub>	T <sub>40</sub>	T <sub>50</sub>	T <sub>60</sub>	T <sub>90</sub>	T <sub>120</sub>	T <sub>140</sub>	T <sub>Poly</sub>	T <sub>ind</sub>
132-2935	-1	0.5	2	7	11	16	33	54.5	60.5	50 min	20 sec
132-2936	-1	0	1	6	9	14	31	54	61	-11-	40 sec
132-2937	0	1	2	6	9	14.5	31	55	62	-11-	6 min
132-2938	0	2	4	8	12	17	34	58	64.5	-11-	30 sec

The rates for these products was very slow but the desired low peak temperatures were achieved. It is hoped that drying (microwave, uv, standard) will activate residual thermals.

The above were all dried on FBD as a standard. Microwaved both with and without V50. The solution used for V50 was 3.5/16.6 w/w (V50 : H<sub>2</sub>O). Results:-

Poly N°	Res Acmt	Visc 1	Visc 2	Solubility	Treatment
132-2935	0.25%	24	25	F/P	None
132-2935A	62 ppm	T-T-L	T-T-L	F/G	Micro + V50 (5ml) *
132-2936	0.11%	19	20	F/G	Microwaved
132-2936	>0.4%	23	23	F/G	None
132-2936A	57 ppm	T-T-L	T-T-L	F/P	Micro + V50 (5ml) *
132-2936B	586 ppm	17	17	F/P	Microwaved
132-2937	0.24%	24	24	F/G	None
132-2937A	59 ppm	T-T-L	T-T-L	F/P	Micro + V50 (5ml) *
132-2937B	556 ppm	17	17	F	Microwaved
132-2938	>0.4%	23	23	F/G	None
132-2938A	134 ppm	T-T-L	T-T-L	F	Micro + V50 (5ml)
132-2938B	643 ppm	16	16	F	Microwaved

I declare that this work was done by me

*afner*

I declare that this work was explained to me and that I have understood it

*P. H. H. H.*

Project No 1542

## Reduction of Residual Monomer

four standard monomers were initiated to treat in different ways. The polymerisation data is shown below.

Poly. N°	KBrO <sub>3</sub>	Na <sub>2</sub> S <sub>2</sub> O <sub>4</sub>	T <sub>0</sub>	T <sub>10</sub>	T <sub>20</sub>	T <sub>40</sub>	T <sub>60</sub>	T <sub>80</sub>	T <sub>90</sub>	T <sub>end</sub>
132-2939	3.5	7	0.5	3	6	18	58	88	90	10 sec
132-2940	3.5	7	0	2.5	6	17	49	87	90	10 sec
132-2941	3.5	7	-0.5	1.5	4	15	45	85	90	10 sec
132-2942	3.5	7	0	3	6	17	52	85	90	10 sec

To verify that it was the V50 and not the low solids which lead to the earlier results (see page 47) <sup>above</sup> standards were to be dried using V50 as a spray on solution.

① Fluid bed dried - no V50

FPR N°	Res Acum	Visc (cP)	Sol
132-2939	754 ppm	22	F/G
2940	876 ppm	22	F/G
2941	830 ppm	24	F/G
2942	957 ppm	23	F/G

② Fluid bed dried + V50 (1.5 ml of a 3.5 g in 16.5 g H<sub>2</sub>O) <sup>or 1 ml</sup>

FPR N°	Res Acum	Visc (cP)	Sol
132-2939 - A	362 ppm	19	F/G
132-2940 - A	328 ppm	20	F/G
132-2941 - A	333 ppm	19	F/G
132-2942 - A	382 ppm	20	F/G

I declare that this work was done by me

*afmer*

I declare that this work was explained to me and that I have understood it

*P. J. J. J.*



CONTROL No.

ROLL

START

END

IND?

Project No. 1342

③ Fluid bed dried (glass head) + UV light and containing V50.

FPR N°	Res Acn	Visc (cP)	Sol	
132-2939-B	213ppm	19	F/G	1ml V50 sol <sup>n</sup>
132-2940-B	123ppm	17	F/G	1.5ml V50 sol <sup>n</sup>
132-2941-B	178ppm	20	F/G	1ml V50 sol <sup>n</sup>
132-2942-B	161ppm	19	F/G	1.5ml V50 sol <sup>n</sup>

[132-2940-B-1 116ppm ♥]

④ Microwave drying with the standard samples (132-2939→2942) containing V50.

FPR N°	Res Acn	Visc	Sol	
132-2939-C	90ppm	T-T-L	F/G	1ml V50 sol <sup>n</sup>
132-2940-C	55ppm	T-T-L	F/G	1.5ml V50 sol <sup>n</sup>
132-2941-C	111ppm	T-T-L	F/G	1ml V50 sol <sup>n</sup>
132-2942-C	115ppm	T-T-L	F/G	1.5ml V50 sol <sup>n</sup>

Visc Spec. 17-25-37

It appears that V50 in conjunction with microwaving is the best option for reducing residual<sup>s</sup> at the moment. Work must be done to improve visc.

It was decided to investigate the possibility of reducing residual acrylamide on cationic powder products as well as anionic ones. <sup>2.0</sup> Zetag 92<sup>monomer</sup> was prepared and initiated <sup>following</sup>. The polymers were produced.

I declare that this work was done by me

*after*

I declare that this work was explained to me and that I have understood it

*f. Heger*

## ARCHIVE DETAILS

CONTROL No.

ROLL START END INI

Project No. 1542

FPR N°	KBrO <sub>3</sub>	Na <sub>2</sub> SO <sub>3</sub>	T <sub>0</sub>	T <sub>10</sub>	T <sub>40</sub>	T <sub>60</sub>	T <sub>80</sub>	T <sub>120</sub>	$\epsilon_{max}$	$\epsilon_{end}$
132-2942	3	6	1	4.5	8	14	23	73	83	7 mins
132-2943	3	6	-1	2.0	5	9	16	46	82	
132-2944	3	6	0	3.0	7	13	22	74	84	8 mins
132-2945	3	6	0	3.0	6	11	27	38	84	16 mins

Visc spec. 130-170-210

@ 30 s<sup>-1</sup>

## RESULTS

① Fluid bed dried no V50

FPR N°	Res Acrylamide	Viscosity (cP)	Sol.
132-2942	798	218 216	F
132-2943	911	215 209	F
132-2944	913	215 210	F
132-2945	975	214 212	F/P

Visc spec 130-170-210

② Microwave + V50 <sup>spray</sup> (3.5 = 16.5g  $\Rightarrow$  ~3000ppm sol<sup>n</sup> on 100g wet gel)

FPR N°	Res Acrylamide	Viscosity (cP)	Sol.
132-2942 A	60	T-T-L	F
132-2943 A	75	T-T-L	F
132-2944 A	89	T-T-L	F
132-2945 A	75	T-T-L	F/P

~~Microwaving with V50 was thought to be necessary as all previous work on this project has used M351. Therefore microwaving alone may reduce residual acrylamide in the catenane powder products.~~

I declare that this work was done by me

A. Hargrave

I declare that this work was explained to me and that I have understood it

A. Hargrave

## ARCHIVE DETAILS

CONTROL No.

ROLL START

END

IND?

Project No. 1542

③ Microwave → V50 spray lower ppm on ~~the~~ wet polymer 250ppm

FPR N°	Res. Acum	Viscosity		Sol	
132-2942B	331	136	133	F/P	1.7ml (283ppm)
132-2943B	380	145	141	F/P	1.5ml (250ppm)
132-2944B	463	162	165	F/P	1.7ml (283ppm)
132-2945B	371	142	142	F/P	1.5ml (250ppm)

④ FBD dry + UV + V50 (250ppm)

FPR N°	Res Acum	Viscosity		Solubility	
132-2942C	332	193	189	Fair	0.75ml (250ppm)
132-2943C	389	179	174	F/P	1.5ml (250ppm)
132-2944C	416	196	194	F/P	0.75ml (250ppm)
132-2945C	537	199	199	Fair	1.5ml (250ppm)

Visc spec 130-170-210 cP

@30s<sup>-1</sup>Previous sol<sup>n</sup> of V50 = 3000ppm3.5g in 16.5 → 21.2% sol<sup>n</sup>

(3.5 → 20mls)

1.5mls on 100g polymer

$$\text{ppm} = \frac{\text{wt added} \times 1 \times 10^6 \times \text{sol}^n \text{ str}}{100 \times 100}$$

$$= \frac{3.5 \times 100}{100 \times 100} = 3500 \text{ ppm}$$

For 250ppm a 1.66% sol<sup>n</sup> is req<sup>d</sup> (DIW).

(0.8335 g of V50 ⇒ 50g)

It was decided to drop the levels of V50 to 125ppm, ~~so~~ this would allow a comparison with the higher levels. Also as

I declare that this work was done by me

after

I declare that this work was explained to me and that I have understood it

M. B. B.

Project No.

V50 is expensive the lower the <sup>effective</sup> level the better.

In spec viscosity material has been produced via the microwave drying route. The wet gel was dried for less time than ~~on~~ on previous occasions (~20 mins 10 mins @ 450W and 10 mins @ 150W) and was allowed to stand. "Cooking" continued ~~and~~ and the gel dried without further microwaving. The solubility of the powder may have been affected slightly by the microwave process but the solubility <sup>of the standards</sup> was ~~not~~ fair only. It may be possible to reduce the microwave cooking time as some parts of the gel appeared scorched after 10 mins @ 450W. Reducing the time spent drying @ 450W or using only 150W may be better for controlling viscosity and solubility.

Again results show that high levels of V50 and microwave treatment has reduced the residual monomer level to  $\leq 100\text{ppm}$ . The lowest ~~level~~ level of V50 (250ppm) was effective in that residual monomer was reduced from 8-900ppm - 3-400ppm.

I declare that this work was done by me

*apm*

I declare that this work was explained to me and that I have understood it

*J. Morgan*

Page No. 68

## ARCHIVE DETAILS

CONTROL No.

ROLL

START

END

Project No. 1542

Reduction of Residual Acrylamide

The following polymers 129-2937

→ 2940

were used to dry in the microwave in a number

of ways:-

FPR No	Comments
129-2937A	std size chip 100ppm V50 microwave 1/2 dry 100ppm UV FBD
(so 2937B	1/2 " " " "
2937C	std size 500ppm " " 500ppm " "
2937D	Small size " " " "
2937E	3000ppm V50 std chip size " " " "

129-2938A	3000ppm V50 standard chip size
2938B	1500ppm V50 1/2 " " "
2938C	1500ppm V50 standard chip size
2938D	Microwaved no V50 std size
2938E	" " 1/2 " "

129-2939A	3000ppm V50 standard chip size microwaved
2939B	750ppm V50 " " " "
2939C	750ppm V50 - 1/2 " " " "
2939D	Microwave 10mins @450W (No V50) → 750ppm V50 (blender)
2939E	" " " "

129-2940A	3000ppm V50 microwaved
2940B	500 1000ppm V50 micro 10mins → UV light / drying
2940C	500 2000ppm V50 " " " "
2940D	Microwave 10mins then 500 1000ppm V50 UV drying
2940E	" " 2000ppm " "

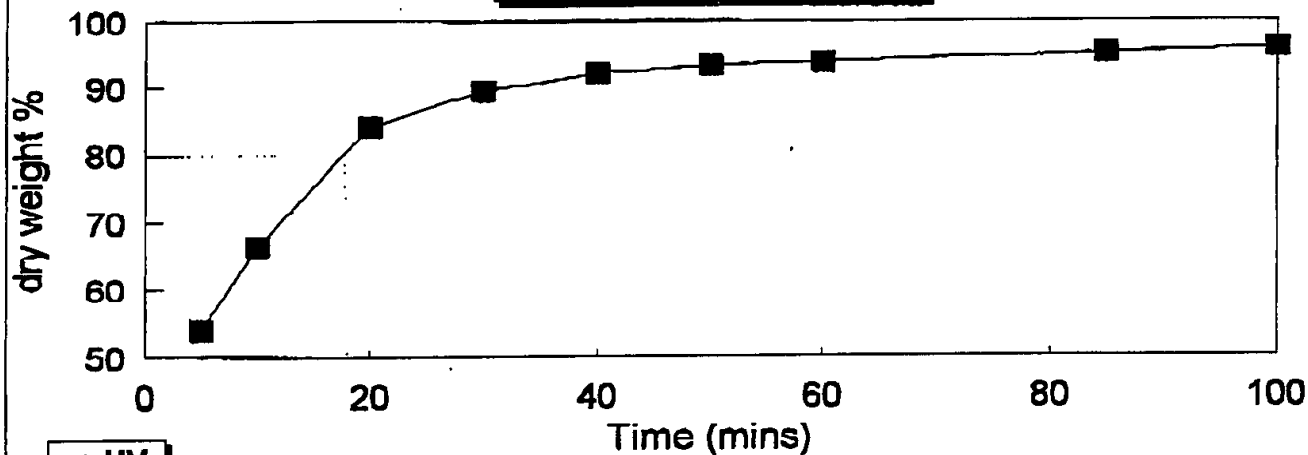
I declare that this work was done by me

updater

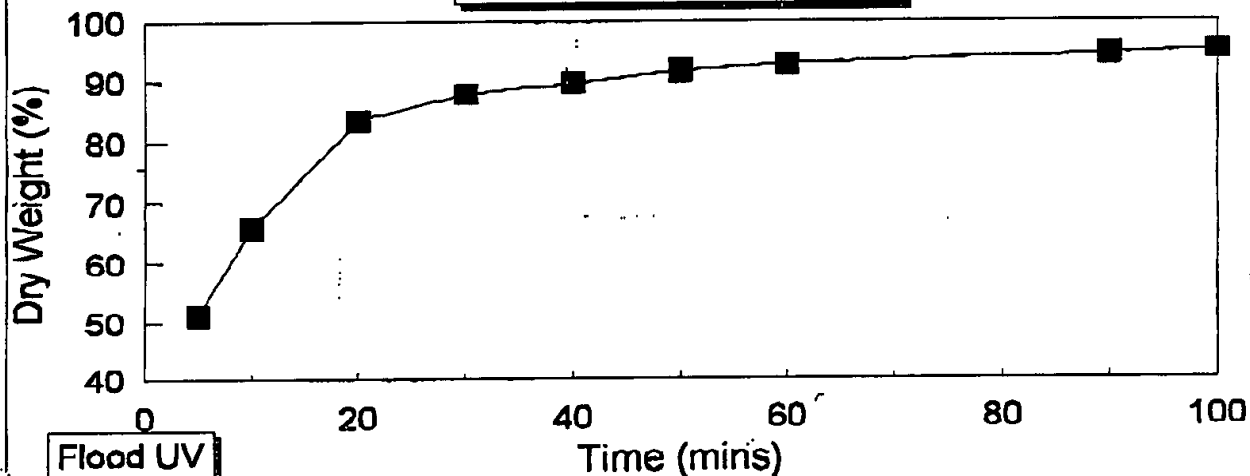
date .

I declare that this work was explained to me and that I have understood it

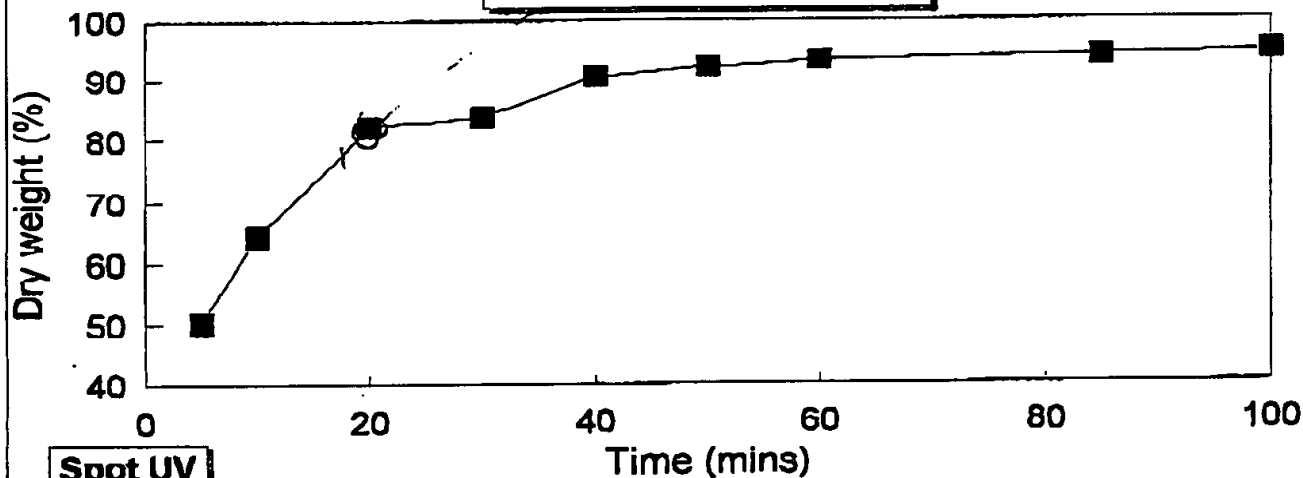
### Drying curve

**no UV**

### Drying Curve

**Flood UV**

### Drying Curve

**Spot UV**

ARCHIVE DETAILS

CONTROL No. ROLL START END IND7

--	--	--	--	--

Project No.

Results

FPR No.	Visc 1	Visc 2	Solubility	Residual Monomer
<del>129</del> 2937A	TTL	TTL	F/g	32
2937B	"	"	F/g	57
2937C	<del>B</del> TTL	<del>14</del> TTL	F/g	57
2937D	15	14	F/g	144
2937E	13	TTL	F/g	98
2938A				
2938B				
2938C				
2938D				
2938E				
2939A				
2939B				
2939C				
2939D				
2939E				
2940A				
2940B				
2940C				
2940D				
2940E				

\*129 - 2940 B 500ppm V50 spray Microwave 10mins @ 450W UV std  
 129 - 2940 C 300ppm V50 spray " " " " " "  
 129 - 2940 C 500ppm V50 spray " " " " UV small.  
 129 - 2940 D 500ppm V50 spray " " " " no uv std  
 129 - 2940 E 500ppm V50 spray " " " " no uv small

I declare that this work was done by me

*cf/over*

date

I declare that this work was explained to me and that I have understood it

*P. J. J. J. J.*

date

## ARCHIVE DETAILS

CONTROL No.

ROLL

START

END

IND7

Project No. 1517

Having established several possibilities which would facilitate the reduction of residual monomer to  $\leq 100$  ppm levels an experiment was planned which would allow the comparison of a number of variables. These were:-

Levels of initiator V50, VAO44

Fluid bed drying

Fluid bed drying in conjunction with UV treatment

Microwaving followed by FBD

Microwaving followed by FBD with UV treatment.

V50/VAO44 Levels	FBD	FBD+UV	Micro $3\frac{1}{2}$ min 450W $\rightarrow$ FBD	Micro $3\frac{1}{2}$ min 450W UV
0	A	B	C	D
500	E	F	G	H
1000	I	J	K	L
2000	M	N	O	P
3000	Q	R	S	T

Production plant polymer (M333) was obtained to ensure a constant source of product. "F" above for example has 500ppm <sup>V50/VAO44</sup> sprayed onto the surface and dried in a fluid bed drier (glass head) under ultra violet light. "T" was sprayed with 3000ppm V50/VAO44 - microwaved for  $3\frac{1}{2}$  minutes @ 450 then FBD in a glass head under ultra violet light.

I declare that this work was done by me

afroher

I declare that this work was explained to me and that I have understood it

f. [signature]



## ARCHIVE DETAILS

CONTROL No.

ROLL START END IN

Project No. 1542

RESULTS		V50		Sol	Res. Acum	FPR No
		Visc 1	Visc 2			
A	1	25	23	G	481	132-2999
B	2	29	27	G	427	3000
C	3	29	28	F/a	498	3001
D	4	28	27	F/a	385	3002
E	5	29	26	G	297	3003
F	6	26	24	G	189	3004
G	7	20	19	G	340	3005
H	8	19	19	F/a	152	3006
I	9	22	22	G	306	3007
J	10	23	23	F/a G	268	3008
K	11	14	15	G	268	3009
L	12	18	17	G	114	3010
M	13	24 24	22 23	G	162	3011
N	14	23 23	22 21	G	142	3012
O	15	17 16	16 15	G	136	3013
P	16	16 15	15 14	G	79	3014
Q	17	27	24	G	156	3015
R	18	23	23	G	128	3016
S	19	15	15	G	123	3017
T	20	14	13	G	163*	3018

\* Approx due to interferences on the chromatogram

Spec 25-33-40

19/3/98. The following were retested for viscosity to check if viscosity fell over time as in sulphate treatment these results are shown alongside the original results but are highlighted.

I declare that this work was done by me

I declare that this work was explained to me and that I have understood it

## ARCHIVE DETAILS

CONTROL No. ROLL START END IND?

Project No. 1547

Results	Visc 1	Visc 2	Sol.	Res. Acum (ppm)	FPR No.
A1	25	23	G	481	132-2999
B1	29	27	G	427	3000
C1	29	28	F/G	498	3001
D1	28	27	F/G	385	3002
E1	27	25	G	490	3019
F1	23	23	F/G	212	3020
G1	18	17	G	331	3021
H1	15	15	G	94	3022
I1	25	24	G	356	3023
J1	22	23	G	161	3024
K1	14	14	F/G	291	3025
L1	19	18	F/G	77	3026
M1	26 24	24 -*	G	326	3027
N1	23 21	23 -*	G	155	3028
O1	14 13	14 -*	G	106	3029
P1	TTL TL	14 -*	F/G	43	3030
Q1	24	25	G	210	3031
R1	22	22	G	101	3032
S1	13	TTL	G	112	3033
T1	13	TTL	F/G	43	3034

Spec 25-33-40

\* Not enough sample remained to test in duplicate

I declare that this work was done by me

of her

I declare that this work was explained to me and that I have understood it

f. [Signature]

## ARCHIVE DETAILS

CONTROL No.

ROLL

START

END

Project No. 1542

The Reduction of Residual  
Monomer

4 polymers were initiated to give poor solubility & high molecular weight product. M333 were shot non-reverse balance and at low initiator levels.

The following were initiated:-

FPR No	KBrO <sub>3</sub>	Na <sub>2</sub> SO <sub>3</sub>
132-3055	1	2
3056	1.5	3
3057	2	4
3058	2.5	5

No VAO<sub>4</sub> was included as a slow rate was required. The polymerisation details were as follows.

FPR No	T <sub>0</sub>	T <sub>20</sub>	T <sub>40</sub>	T <sub>60</sub>	T <sub>max</sub>	t <sub>pdy</sub>	End
132-3055	0	4.5	8	17	84	380	
3056	-2	4	8	17	86	300	
3057	0	4.5	10	39	86	210	
3058	-1	3.5	12	85	85	180	

The rates for the above were slow and the results for viscosity + solubility were as follows.

I declare that this work was done by me

*after*

I declare that this work was explained to me and that I have understood it

*Libby*

ARCHIVE DETAILS

CONTROL No. ROLL START END IND?

--	--	--	--	--

Project No.

FPR No.	Viscosity	Solubility	Residual Acn
132-3055	31.30	F	499
3056	31.30	F	561
3057	35.34	F	475
3058	31.30	F	428

The above four polys were treated in a number of ways as follows:-

132-3055 A	{	Microwaved in VAO <sub>4</sub>
3056 A		
3057 A		
3058 A		

3 1/2 minutes

132-3055 B	{	2500ppm VAO <sub>4</sub> + microwaved 3 1/2 minutes
3056 B		
3057 B		
3058 B		

132-3055 C	{	2500ppm VSO
3056 C		
3057 C		
3058 C		

132-3055 D	{	2500ppm VSO + uv dried
3056 D		
3057 D		
3058 D		

I declare that this work was done by me

*apner*

I declare that this work was explained to me and that I have understood it

*L. L. L.*

## ARCHIVE DETAILS

CONTROL No.

ROLL

START

END

IND

Project No.

The results were as follows:

FPR No.	Visc		Solubility	Res Acm
132-3055A	22	21	F	479
3056A	34	37	F/G	443
3057A	36	29	F/G	496
3058A	29	28	G	428
3055B	19	18	F/G	132
3056B	18	18	F/G	89
3057B	19	18	F/G	169
3058B	18	18	G	152
3055C	16	15	G	153
3056C	16	15	G	154
3057C	17	16	G	166
3058C	16	16	G	226
3055D	20	19	F/G	99
3056D	26	23	G	338
3057D	27	23	G	196
3058D	24	23	G	102

M333 spec 25-33-40cP

The above results show that the treatment of @ 250s<sup>+</sup>  
the poor ~~sol~~ solubility gel has again reduced the viscosity  
but not within specification. The uv light treatment has  
reduced viscosity (~250cpm 150) but not to the same extent  
as in microwave treatment. The application of foil to the  
glass drying head hopefully will allow more uv light to reach  
and react on the surface of the gel chip. Benzil dimethyl-  
ketol (uv initiator) will be tried.

I declare that this work was done by me

A. P. H.

I declare that this work was explained to me and that I have understood it.

L. H. G.

## ARCHIVE DETAILS

CONTROL No. ROLL START END IND?

Project No. 1542.

Reduction of Residual Acrylamide

A set of experiments were designed which would allow the comparison of particle size, uv light intensity and V50 treatment of 400g of production ME10 was dried in each instance on a FBD in the glass drying head.

The following experiments were to be carried out.

Intensity	Thermal (ppm)	V50	Particle Size	FPR. No.
Low	0		Small	142-087
HIGH	0		"	142-093
L	500		"	142-090
H	500		"	142-094
L	1000		"	142-091
H	1000		"	142-097
L	<del>1000</del>		Large	142-100
H	0		"	142-102
L	500		"	142-103
H	500		"	142-105
L	1000		"	142-106
H	1000		"	142-096
0	0		Small	142-079
0	0		Large	142-080
0	500		Small	142-083
0	500		Large	142-084
0	1000		Small	142-085
0	1000		Large	142-086

I declare that this work was done by me

*afmer*

I declare that this work was explained to me and that I have understood it

*hbs*

ARCHIVE DETAILS

CONTROL No.

ROLL START

END

IND

Project No.

High intensity light was achieved using two ~~spot~~ <sup>from</sup> UV bulbs with low intensity one bulb.

Small particle size ~~2.8mm~~  $< 4mm > 2.8mm$

large particle size  $> 4mm$

Wet gel chip was lubricated and sieved to achieve the desired particle size. V50 was added to the gel chip as it was blended.

Drying temperature  $85^{\circ}C$

Blower speed 7.5

Samples of the drying gel were removed from the bulk sample on the following times.

T=0

5 A

10 B

15 C

20 D

30 E

40 F

50 G

60 H

Dry weight and residual acrylamide were measured. Residual acrylamide was corrected to 100% dry weight.

I declare that this work was done by me

*after*

I declare that this work was explained to me and that I have understood it

*[Signature]*

ARCHIVE DETAILS				
CONTROL No.	ROLL	START	END	IND?

Project No. 1542

RESULTS

FPR No.	A	B	C	D	E	F	G	H
142-079	1496	1340	1396	1250	1325	1300	1396	1279
080	<sup>T=0</sup> 1130	<sup>A</sup> 1147	<sup>D</sup> 1304	<sup>C</sup> 1252	<sup>E</sup> 1300	<sup>F</sup> 1338	<sup>G</sup> 1094	<sup>H</sup> 1087
083	3226	3165	3377	3203	3128	2938	3135	3128
084	2667	2496	2708	2604	2678	3482	2588	2668
085	3257	3198	3445	3140	2791	3034	2817	2781
086	3203	3014	2978	2860	3091	2932	2685	3092
087	<sup>T=0</sup> 2600	2578	2613	2788	2634	2088	1724	1469
090	1369	1771	1221	1253	1273	1064	819	727
091	1251	1289	1380	1408	1296	1220	1088	1081
093	147	1431	1425	1230	992	643	577	527
094	<sup>A</sup> 1113	<sup>B</sup> 1031	<sup>C</sup> 928	<sup>D</sup> 964	<sup>E</sup> 551	<sup>F</sup> 418	<sup>G</sup> 429	<sup>H</sup> 615
096	1226	1171	1120	969	743	510	417	437
097	133	1319	1134	751	444	420	447	443
100	1311	402	1347	1330	1336	1184	1026	893
102	1305	1452	1311	1323	1332	1067	779	730
103	<sup>A</sup> 1109	<sup>B</sup> 1309	<sup>C</sup> 1174	<sup>D</sup> 1169	<sup>E</sup> 1038	<sup>F</sup> 813	<sup>G</sup> 824	<sup>H</sup> 680
105	1327	1284	1098	830	597	498	600	431
106	1275	1332	1230	1302	900	774	682	614

~~continued~~

Not all T=0 samples were taken

I declare that this work was done by me

*afner*

I declare that this work was explained to me and that I have understood it

*h. h. h.*



ARCHIVE DETAILS

CONTROL No. ROLL START END IND?

ject No. 2006

78 Bonson - F.E.D.L.

A factorial experiment has been designed which will allow comparison of a number of factors. This initial screening design will be developed further and drying conditions determined.

pt. No.	V50	VACU	Quantities	Quantities	Liquid	U.V.	Micro	Particle Size	9	10
1-200	X	X	X	X	X	X	✓	Small	✓	✓
1-201	X	X	X	✓	✓	X	✓	Large	X	✓
1-202	✓	X	X	✓	✓	✓	X	Small	✓	X
1-203	✓	X	X	X	X	✓	✓	Large	X	X
1-204	X	✓	X	✓	X	✓	✓	Small	✓	X
1-205	X	✓	X	X	✓	✓	✓	Large	X	X
1-206	✓	✓	X	✓	X	X	✓	Small	✓	✓
1-207	X	X	✓	X	✓	✓	✓	Large	X	✓
1-208	X	X	✓	✓	X	✓	X	Small	✓	✓
1-209	✓	✓	X	✓	X	X	✓	Large	X	✓
1-210	X	✓	✓	✓	✓	X	✓	Small	X	X
1-211	X	✓	✓	✓	✓	X	✓	Large	✓	X
1-212	X	✓	✓	✓	✓	X	✓	Small	X	✓
1-213	X	✓	✓	✓	✓	X	✓	Large	✓	✓
1-214	X	✓	✓	✓	✓	X	✓	Small	X	✓
1-215	X	✓	✓	✓	✓	X	✓	Large	✓	✓
1-216	X	✓	✓	✓	✓	X	✓	Small	X	✓
1-217	X	✓	✓	✓	✓	X	✓	Large	✓	✓
1-218	X	✓	✓	✓	✓	X	✓	Small	X	✓
1-219	X	✓	✓	✓	✓	X	✓	Large	✓	✓
1-220	X	✓	✓	✓	✓	X	✓	Small	X	✓

Exposure thermal treatment large particle size > 4.0mm  
high intensity UV Small particle size < 4.0mm  
Microscopic treatment 2000000 (B.D.) 1/2 inch > 2.5mm @ 150 Watts  
will be described at a later date.

I declare that this work was done by me

april 1997

date

I declare that this work was explained to me and that I have understood it

date

## ARCHIVE DETAILS

CONTROL No.

ROLL START

END

IND

Project No. 2006

Bonsai FED 1

The FED described on page 145 was non-plastic and the results are shown in the table below.

FPE Number	Viscosity	Stability	Residual Area	Dry Weight	
142 - 173	<sup>200 S</sup> 29   <sup>500 S</sup> 25 33	F/G	484	522	92.4
174	TTL 9	F	70	76	92.0
175	TTL 10	F	86	98	87.5
201	TTL 12	F	11	108	91.3
202	TTL 14	F	150	169	113.2
203	24/26 33	F/G	533	586	112.1
204	23/25 30	F/G	429	472	112.0
205	26 33	F/G	416	674	112.4
206	24/24 32	F/G	419	479	97.5
207	15 19	F	267	232	90.7
208	TTL 16	F	115	134	106.1
209	25/25 32	G	574	648	112.5
210	23/23 26	F/G	280	235	103.1
211	24/24 31	F/G	51	567	101.3
212	25/25 34	G	611	652	112.5
213	23/23 27	F/G	27	201	113.5

As some of the above results are TIL, non standard test methods were used to get a number for the viscosity.

In order to evaluate the results simple statistics were used.

The graph shown overleaf is an example of the calculations carried out.

I declare that this work was done by me

albert

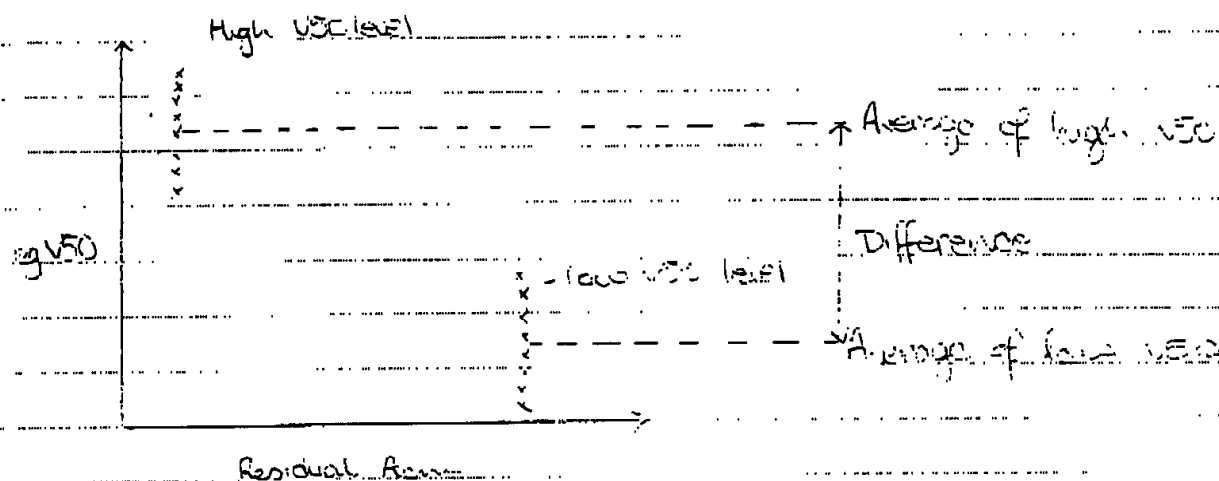
I declare that this work was explained to me and that I have understood it

ARCHIVE DETAILS

CONTROL No. ROLL START END IND?

--	--	--	--	--

Project No.



A high VSC level (in this case 10ppm) residual acrylamide is lower than in the right with low VSC level (0ppm).

This method will allow us to establish which of the 10 factors are having the greatest effect on both residual acrylamide and viscosity. A second F&E can then be designed and conditions optimised further.

The viscosity of the samples was retested at the higher shear rate of 500 s<sup>-1</sup> so that a number could be used in the statistical analysis.

The analysis was carried out for all the variables (V50, VAC, Quanticure B, Quanticure A, liquid Azo, UV, microwave) and also the 'blocking' effects. This Pareto analysis highlights the biggest factors affecting both viscosity and residual acrylamide. The results are shown overleaf.

I declare that this work was done by me

*af/et*

I declare that this work was explained to me and that I have understood it

*[Signature]*

ARCHIVE DETAILS

CONTROL No. ROLL START END IND?

Project No. 2006 Bonsai

We made up the monomer EAC, following the recipe below.  
(reference M2069)

	% @ 100%	Wt taken
Acrylamide	72.614	9019.6736g
Adipic Acid	5	311.15601g
M.B.S	22.368	1960.5458g
Tetralon B	300ppm	18.3g
Water		6990.3246g
AZDN	400ppm	7.32g
Total =		18300g

pH = 3.5

Solids = 34%

We initiated the monomer in the following way:

Initiators	130-	130-	130-	130-	130-	130-
Additives	2331	2332	2333	2334	2335	2336
KBrO <sub>3</sub> 0.135%	4.5	4.5	4.5	4.5	4.5	4.5
Na <sub>2</sub> SO <sub>3</sub> 0.27%	9	9	9	9	9	9
Temperature	Standard	500	2000	Standard	500	2000

All the gels had 30 minutes degas, 2hrs residence and were worked up immediately

I declare that this work was done by me

A. H. Holman

date

I declare that this work was explained to me and that I have understood it

alisher

date

## ARCHIVE DETAILS

CONTROL No.

ROLL START

END

IND?

Project No. 2006 Bonsai

Poly N°	Ind T	T <sub>0</sub>	T <sub>10</sub>	T <sub>20</sub>	T <sub>30</sub>	T <sub>40</sub>	T <sub>50</sub>	T <sub>60</sub>	T <sub>70</sub>	T <sub>80</sub>	T <sub>90</sub>	T <sub>100</sub>	T <sub>110</sub>	T <sub>120</sub>	T <sub>130</sub>
130-2331	60secs	+1	4	6	→	15	23	30	48	66	78	80	80	-	-
130-2332	18 mins	-1	1	2	→	7	12	16	25	30	40	61	72	79	79
130-2333	70secs	0	2	5	→	15	23	31	50	67	85	85	-	-	-
130-2334	60secs	-1 1/2	3 1/2	6 1/2	11	16 1/2	22	→	64	76	76	-	-	-	-
130-2335	3mins 20	-1	0	3	6	11	17	22	→	74	83	82	-	-	-
130-2336	40secs	0	2	5	8	13	19	25	→	68	76	76	-	-	-

We dried the polymers as standard and also with UV.

Results	Exp1 Viscosity	Exp2 Viscosity	Solubility	Res
130-2331 A	200	204	F/G	dried 72
130-2332 A	207	201	G	Std. 967 82
130-2333 A	190	191	G	(no 95
130-2334 A	224	216	F/G	UV light 136
130-2335 A	198	199	F/G	95
130-2336 A	212	209	G	97

\*reaction  
group  
& moieties  
lost.

Results	Exp1 Viscosity	Exp2 Viscosity	Solubility	Res
130-2331 A				dried
130-2332 A				with
130-2333 A				UV
130-2334 A				light (all
130-2335 A				the way
130-2336 A				through) 93

never carried out

130-2331 B	188	189	F/G	dried 95
130-2332 B	188	181	F/G	51
130-2333 B	172	171	F/G	with 20
130-2334 B	170	196	F/G	mins UV
130-2335 B	175	173	F/G	7
130-2336 B	174	172	G	21

I declare that this work was done by me

Amtholman

date

I declare that this work was explained to me and that I have understood it

after

date

Addition of Irgacure

## ARCHIVE DETAILS

CONTROL No. ROLL START END IND?

Project No.

to M333 monomer.

We made up 6 polys of M333 following the recipe below  
ref M2117

Acrylamide	10388.4g
Adipic Acid	54.168g
Urea	54.168g
Tetralon B 300ppm	18.3g
Water	7784.98g
AZDN 450ppm	8.235g

pH 3.5

Solids 29.6%

2 standards	no irgacure
2 @ 3.05g	500ppm Irgacure
2 @ 12.2g	2000ppm Irgacure

We polymerised them as follows:

Initiators & Additives	129- 3266	129- 3267	129- 3268	129- 3269	129- 3270	129- 3271
VAO <sub>44</sub> 1.5%	50	50	50	50	50	50
KBrO <sub>3</sub> 0.36%	12	12	12	12	12	12
Na <sub>2</sub> SO <sub>3</sub> 0.15%	5	5	5	5	5	5
Irgacure	0	500	2000	0	500	2000

We gave all the polys 30 mins degas and  
4 hrs residence.

I declare that this work was done by me

A. Holman

date

I declare that this work was explained to me and that I have understood it

af. hex

date

Addition of Irradiation  
to m333 monomer

Project No.

ARCHIVE DETAILS				
CONTROL No.	ROLL	START	END	IND7

Poly N°	Ind T.	T <sub>5</sub>	T <sub>15</sub>	T <sub>20</sub>	T <sub>30</sub>	T <sub>40</sub>	T <sub>50</sub>	T <sub>60</sub>	T <sub>70</sub>	T <sub>80</sub>	T <sub>90</sub>	
129-3266	1m 30	+1	3	10	20	33	52	85	83	-	-	
129-3267	1m 10	+1 1/2	3	12	22	38	76	86	86	-	-	← Irq
129-3268	20	+2	4	12	22	36	68	87	87	-	-	← Irq
129-3269	5m 40	+1	2	8	13	21	35	55	80	84	83	
129-3270	1m 50	0	1	8	14	24	42	82	85	85	84	← Irq
129-3271	40	+1	3	10	18	29	55	84	84	84	83	← Irq

Results

\* Residual Acn.

Dried Standard

Poly N°	Exp1 Viscosity	Exp2 Viscosity	Sol.	Res Acn	
129-3266 A	30	26	F/G	532 ppm	0
129-3267 A	26	25	F/G	552 ppm	500
129-3268 A	27	28	F/G	639 ppm	200
129-3269 A	31	29	F/G	613 ppm	0
129-3270 A	30	27	F/G	580 ppm	500
129-3271 A	31	28	F/G	687 ppm	0

Dried 20min stiv.

Poly N°	Exp1 Viscosity	Exp2 Viscosity	Sol.	Res Acn.	
129-3266 B	24	23	F/G	412 ppm	0
129-3267 B	24	23	F/G	38 ppm	500
129-3268 B	25	23	F/G	32 ppm	200
129-3269 B	28	25	F/G	36 ppm	0
129-3270 B	27	25	F/G	48 ppm	500
129-3271 B	25	24	F/G	33 ppm	000

I declare that this work was done by me

Stollman

date :

I declare that this work was explained to me and that I have understood it

Alfner

date

CONTROL No.

ROLL START

END

II

Project No.

Anionic monomer

We made up 4 polys of BTU monomer following the recipe below.

Acrylamide	3610g
Acrylic Acid	1606g
Caustic	1482g
Urea	73.2g
Terralon B	12.2g
Water	5416g
AZDN	3.05g
ACVA	3.05g

To 2 polys we added 500 ppm of Irgacure 2959 and the other 2 had none.

500 ppm = 3.05g Irgacure 2959

We initiated them as below:

Initiators + Additives	129-3427	129-3428	129-3429	129-3430
tBHP	3.5	3.5	3.5	3.5
Na <sub>2</sub> SO <sub>3</sub>	7	7	7	7
I2959	0	0	500	500

They were given 10 mins degas + 2 hrs residence.

I declare that this work was done by me

Stelman

date

I declare that this work was explained to me and that I have understood it

afaner

date



Reaction inAnionic monomer

Project No.

## ARCHIVE DETAILS

CONTROL No.

ROLL

START

END

IND?

Poly N°	Ind T	T0	T5	T10	T20	T40	T50	T60
129-3427	10s	-3	32	43	58	69		68
129-3428	10s	-2	32	44	59	72		72
129-3429	10s	-3	30	43	58	70		70
129-3430	12s	-1	32	44	59	70		70

We dried the polys as follows:

- (A) 200g dried in glass head (no UV)  
 (B) 200g dried in glass head + UV for first 20 mins.

Results

Poly N°	Exp1 VIS	Exp2 VIS	Sol	Ras Acn.
129-3427A	40	40	F/G	168
129-3427B	39	40	F/G	56
129-3428A	40	40	F/G	122
129-3428B	39	39	F/G	43
129-3429A	41	42	F/G	386
129-3429B	37	37	F/G	27
129-3430A	37	36	F/G	96
129-3430B	39	39	F/G	22

I declare that this work was done by me

Holman

date

I declare that this work was explained to me and that I have understood it

alder

date

## ARCHIVE DETAILS

CONTROL No.

ROLL START

END

IND?

Project No.

We made up 4 polys of P182 (see recipe below)  
ref M2201

Acrylamide		5175g
Adipic Acid		207g
MBS		1221g
Tetralon B	300ppm	12.2g
Water		5044g
AZDN	500ppm	6.1g
ACVA	100ppm	1.22g

2 polys had  
500ppm T2959  
= 3.05g

Initiators + Additives	130 - 2455	130 - 2456	130 - 2457	130 - 2458	
KBrO <sub>3</sub>	4	4	4	4	They had 1/2 hr degas + 2 hrs residence
Na <sub>2</sub> SO <sub>3</sub>	8	8	8	8	
T2959	0	0	500	500	

Poly N°	Ind T	T <sub>0</sub>	T <sub>5</sub>	T <sub>10</sub>	T <sub>20</sub>	T <sub>30</sub>	T <sub>40</sub>	T <sub>50</sub>	T <sub>60</sub>	T <sub>70</sub>	T <sub>80</sub>	T <sub>90</sub>	T <sub>100</sub>	T <sub>110</sub>	T <sub>115</sub>	T <sub>120</sub>	T <sub>125</sub>
130-2455	25s	+2	3	3½	6	9	13	17	24	30	40	50	85	86	86	85	-
130-2456	6m28	+2	2	3	5	7	11	14	19	25	32	42	60	86	87	87	-
130-2457	FTP	+3	4	4	6	7	9	11	15	18	23	28	36	44	54	73	86
130-2458	6m59	+2	3	4	5	8	11	15	22	27	35	46	76	85	85	85	-

I declare that this work was done by me

Staelman

date

I declare that this work was explained to me and that I have understood it

Aloher

date

Irregular In  
P182.

Project No.

ARCHIVE DETAILS				
CONTROL No.	ROLL	START	END	IND?

Results

Poly Number	Exp1 Vis	Exp2 Vis	SDI	Res Acn	LSLC	
					Soft	xlinked
130-2455 A	183	187	G	837	1 0 1	0 0 0
130-2455 B	188	191	G	784	0 0 0	0 0 0
130-2456 A	198	194	G	789	0 5 3	0 0 0
130-2456 B	187	189	G	569	0 0 2	0 0 0
130-2457 A	181	177	G	813	0 2 1	0 0 0 *
130-2457 B	177	181	G	78	0 0 0	0 0 0
130-2458 A	189	180	G	710	2 2 2	0 0 0
130-2458 B	176	184	G	85	1 0 2	0 0 2 *

~~a few~~ undissolved lumps.

I declare that this work was done by me H. Holman date .

I declare that this work was explained to me and that I have understood it Donner date .

BZX

(A235)

Project No.

## ARCHIVE DETAILS

CONTROL No.

ROLL START END

IND?

4 polys of BZX were made following the  
recipe below ref M2203

Acrylamide	5565.3g
Acrylic Acid	1196.2g
Caustic	1173.4g
TetralonB	12.2g
Water	4252.8g
A2DN	4.88g
ACUA	4.88g

pH 6.9  
32% solids

1 poly = 0ppm I2959  
1 poly = 100ppm I2959  
1 poly = 250ppm I2959  
1 poly = 500ppm I2959

They were shot as below (10mins degas, 2hrs residence  
- worked up immediately)

Initiators + Additives	129- 3457	129- 3458	129- 3459	129- 3460
Hypod 0.9%	30	30	30	30
tBHP 0.18%	6	6	6	6
N <sub>2</sub> SO <sub>3</sub> 0.36%	12	12	12	12
I2959	0	100	250	500

Poly Number	Ind Time	T0	T5	T10	T20	T30	T40	T50
129-3457	25 secs	0	24	35	67	87	88	87
129-3458	27 secs	-1	20	31	62	87	87	86
129-3459	22 secs	-1	22	35	74	88	88	87
129-3460	20 secs	0	15	31	61	88	89	88

I declare that this work was done by me

M. H. H. H. H. H.

date

I declare that this work was explained to me and that I have understood it

after

date

Ligature In  
BZX

Project No.

ARCHIVE DETAILS				
CONTROL No.	ROLL	START	END	IND?

Results

Poly Number	Exp1 Vis	Exp2 Vis	SOI	ROS ACM
129-3457A	144	no duplicates	G	32
129-3457B	124		G	19
129-3458A	143		G	26
129-3458B	130		G	16
129-3459A	137		G	31
129-3459B	123		G	14
129-3460A	141		G	29
129-3460B	126		G	10

I declare that this work was done by me

Sholman

date .

I declare that this work was explained to me and that I have understood it

apohar

date .

## ARCHIVE DETAILS

CONTROL No. ROLL START END IND?

Project No. 2006

BONGAI - The Reduction of Residual Acrylamide  
on Powder Products

Irradiation 2959 Optimisation FED - see FPR Notebook 76/191-193

The results from the FED are shown below, viscosity, solubility and residual acrylamide were evaluated.

FPR No	Viscosity	Solubility	Res Acry
142-353	21	F/G	695
354	18		447
355	21		238
356	18		216
357	22		940
358	18		472
359	22		408
360	18		274
361	21		354
362	17		197
363	19		158
364	17		106
365	21		346
366	18		260
367	20		172
368	17		105
369	19		243
370	21		220
371	20		267
372	20	✓	333

Viscosity specification  
Organopol 21 (CL2)  
14-18-21 cP

Centre Points

I declare that this work was done by me

A. P. H.

date

I declare that this work was explained to me and that I have understood it

date

ARCHIVE DETAILS

CONTROL No. ROLL START END IND?

--	--	--	--	--

Project No. 200E

Inclusion of D1173

As the use of D1173 has proved successful on project Iconus, it was decided to use it on Bonsai + include it in the monomer mixture. The Darocure was added in methanol during degassing. The standard contained methanol + no D1173. The following polymers were initiated.

Zetac 72

FPR No. Darocure 1173

130-2440	0	* small amounts added incorrectly - polymerised,
130-2441	42*	worked up + treated.
130-2442	84*	

Polymerisation Information

FPR No.	T <sub>0</sub>	T <sub>10</sub>	T <sub>20</sub>	T <sub>40</sub>	T <sub>60</sub>	T <sub>max</sub>	t <sub>ind</sub>	t <sub>poly</sub>
130-2440	3	8	15	45	85	85	10sec	70min
2441	2	6	12	41	86	87	10sec	80min
2442	2	6	13	41	83	84	10sec	80min

45 KB-O<sub>2</sub>

9 N<sub>2</sub>SO<sub>3</sub>

Each of the above received a 30 min N<sub>2</sub> purge time + 2hr oven residence at 85°C. The gels were worked up immediately after oven residence. Each was dried with/without UV treatment (20 mins). Those labelled "A" were given 20 mins UV treatment.

~~130-2440~~

~~130-2440~~ 440 2440A

2441 709 2441A 252

2442 662 2442A 232

I declare that this work was done by me

afonex

date

I declare that this work was explained to me and that I have understood it

date

Page No. 16

## ARCHIVE DETAILS

CONTROL No. ROLL START END IND?

Project No. 2006

Although  $<100\text{ppm}$  had not been achieved, <sup>due to</sup> the low levels of D1173 used, a significant reduction was seen. This work was repeated using higher levels of D1173 (no MeOH).  
M333 (292 not initiated due to quat shortage)

FPR No	D1173
129-3439	0
3440	100
3441	200
3442	500

Initiator levels of 12:5 KBrO<sub>3</sub>:Na<sub>2</sub>SO<sub>3</sub> with 50ppm VAD<sub>4</sub> were used

## Polymerisation Information

FPR No	T <sub>0</sub>	T <sub>10</sub>	T <sub>20</sub>	T <sub>30</sub>	T <sub>40</sub>	T <sub>50</sub>	T <sub>max</sub>	t <sub>ind</sub>	t <sub>dy</sub>
129-3439	0	4	11	28	46	84	85	15s	80
3440	0	4	12	29	55	89	89	40s	80
3441	0	4	11	29	49	84	84	20s	80
3442	0	1	5	14	22	48	85	9m 40	80

Each of above were UV treated <sup>+20mins 'B'</sup> + dried as standards 'A'

FPR No	Visc	Sol	Res. Acn (ppm)
129-3439 A	29	G	479
3439 B	26	G	446
3440 A	29	G	577
3440 B	24	G	110
3441 A	28	G	509
3441 B	25	G	73
3442 A	31	G	587
3442 B	25	G	42

I declare that this work was done by me

A. Jones

I declare that this work was explained to me and that I have understood it



## ARCHIVE DETAILS

CONTROL No. ROLL START END IND?

Project No.

The results were as follows:-

FPR No	Visc	Solubility	Res. Acn
129-3447A	42	Closed	119
B	3940	"	62
3448A	43	"	80
B	38	"	47
3449A	44	"	97
B	38	"	24
3450A	47	"	149
B	38	"	26

Control Drying Curves

In order to establish how quickly the I2959 present in the monomer is able to reduce free acrylamide, drying curves were plotted. Samples were removed from the drying chip and evaluated for dry weight and residual acrylamide. Dry weight corrected residual acn's (95%) are plotted against time, with the corresponding dry wt on a second y-axis.

The graphs shown overleaf are

Sample times (min)

FPR No.

129-3451A No I2959, no UV

0

B No I2959 + UV light

5

C No I2959 + UV - drier no heat

10

129-3452 A As above with 500ppm I2959

15

B

20

C

30

50

I declare that this work was done by me

afher

I declare that this work was explained to me and that I have understood it

--	--	--	--	--

129-3451 A-C



after

**date**

explained to me and that

date

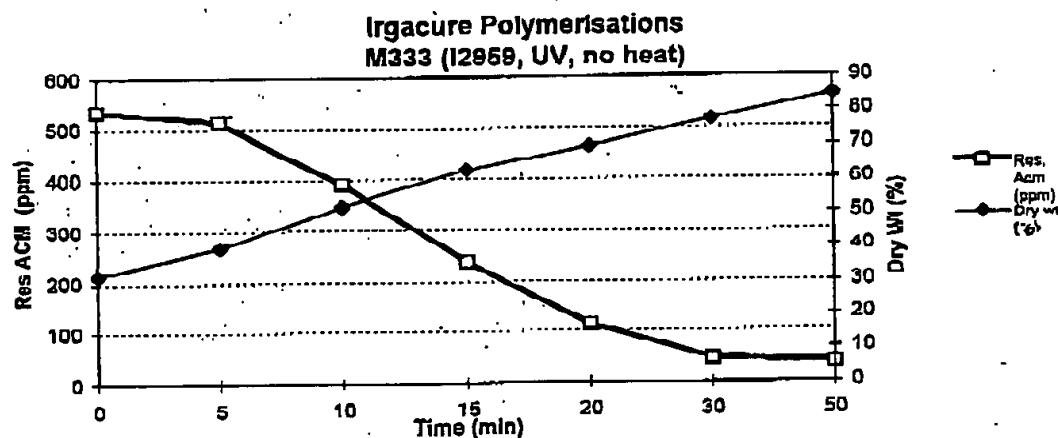
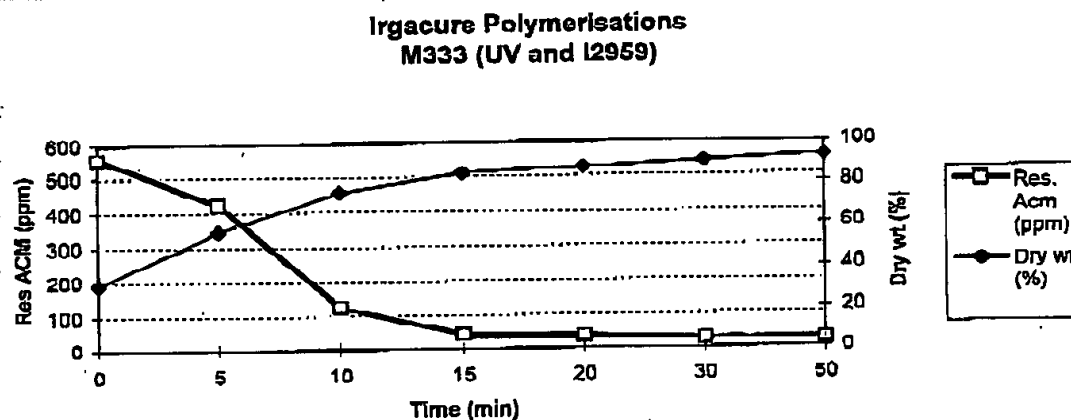
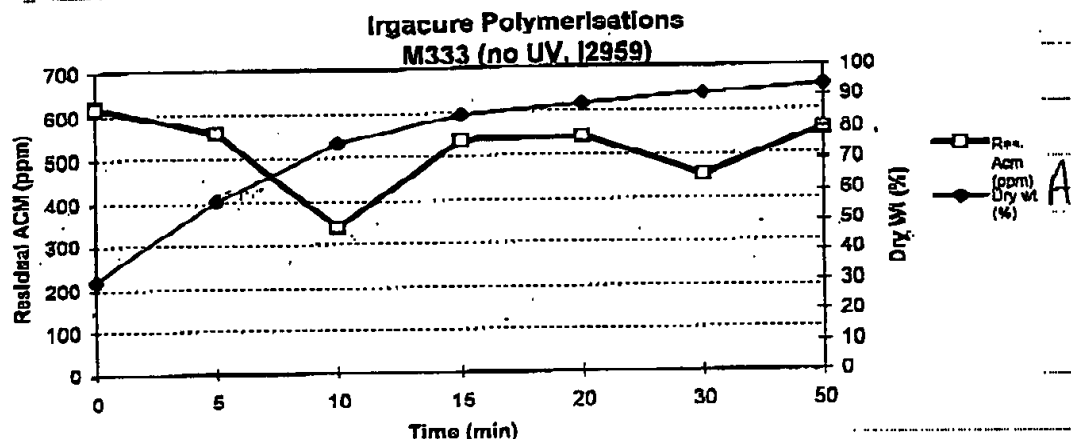
# ARCHIVE DETAILS

CONTROL No. ROLL START END IND?

--	--	--	--	--

Project No.

129-3453-A-C



I declare that this work was done by me

*alex*

date .

I declare that this work was explained to me and that I have understood it

*[Signature]*

date :

ARCHIVE DETAILS

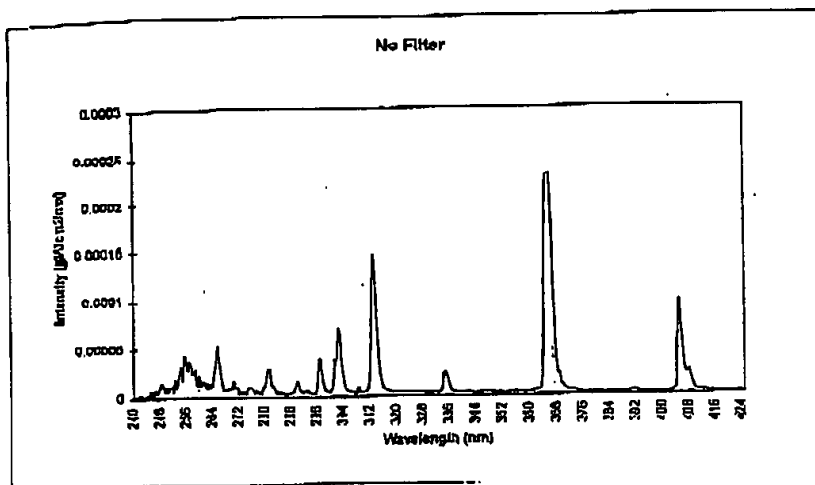
CONTROL No. ROLL START END IND?

--	--	--	--	--

Project No. 2006

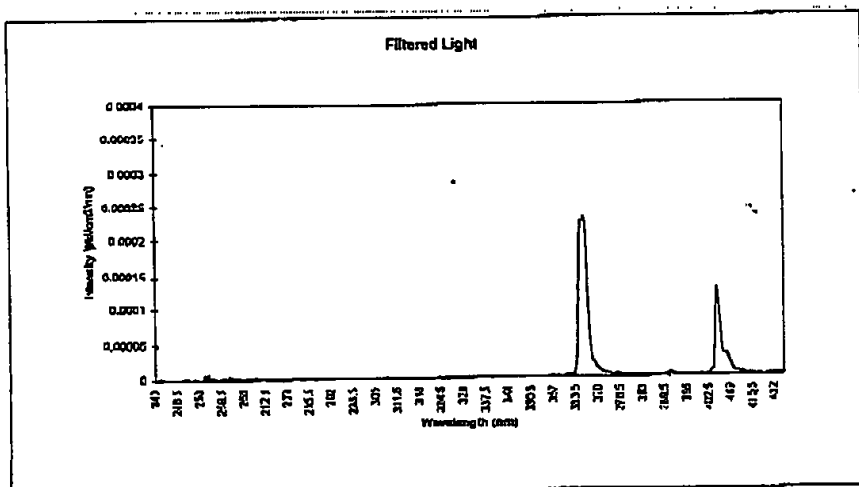
Effect of Lower Wavelength on Viscosity - Residual Acrylamide

In order to investigate the effect of the lower wavelengths on viscosity and residual acrylamide the glass filter was removed from the Starna lamp. The following spectrum was achieved.



Spectrum 1

The lower end of the spectrum is transmitted (the glass filters this region out - see below).



Spectrum 2

I declare that this work was done by me

*OKover*

date .

I declare that this work was explained to me and that I have understood it

date .

ARCHIVE DETAILS

CONTROL No. ROLL START END IND7

Project No.

The intensity of the unfiltered light was adjusted using the variable transformer to give a similar intensity to the filtered light. Glass also reduces intensity.

100g of lubricated gel chip was spread onto a dinner plate and placed under the UV light either with or without a filter. Samples of wet gel were removed after 10, 20, 30 and 40 minutes - the results are as follows:-

FPR No 129-3480 was used

Res. Acn (ppm)

129- Filtered (c) Visc Unfiltered (D) Visc

~~3780~~ C 101 512 96

2 454 71

3 451 64

4 321 39

Standard 129-3480 635 ppm residual acrylamide

All above samples were dry weight corrected to 95%.

The gel which had unfiltered light treatment went very yellow and stuck together even after 10 minutes. Viscosity of the two samples taken after 40 mins was also measured.

FPR No 129-3480C4 was dried and put on to tumble - after 3 hours the solubility was poor with many x-linked lumps.

I declare that this work was done by me

*ofher*

date .

I declare that this work was explained to me and that I have understood it

date .

Project No. 2006

Autocatalytic Work

Standard 292 was manufactured containing no thermal (500) and was degassed over 60mins ( $2 \text{ L/hr}^{-1}$ ) to check the stability of D1173 and I2959 and if autocatalytic would occur. D1173 was added during the first stages of degas. I2959 was in monomer.

7 monomers were made and degassed for 60 mins - the following levels of I29, D1173 + I2959 were used

SG D1173 1.08g/ml

	0	300	500	1000	2000	20000 ppm
ms	0.139	0.231	0.463	0.926	9.26	

None of the D1173 containing monomers 'went off' during degas. This suggests that even at very high levels it would not lead to product autocatalytic with D1173.

Impure 2959

No autocatalytic occurred when I2959 was added to the monomer in the above amounts. This suggests that the photoinitiator is stable at least for 60 mins under fully degassed conditions.

Anionics were also assessed. M1011 was manufactured and the same level of photoinitiators used. Degass time on production for anionic products is less than cations (10mins) so the monomer may be more prone to autocatalytic.

None of the above polymerised after 60 min degas ( $2 \text{ L/min}^{-1}$ ) although the 2000ppm level was very foamy and stringiness was evident after 50mins. The bubblers were swapped to ensure fair comparison.

I declare that this work was done by me

of the

date

I declare that this work was explained to me and that I have understood it

date

TOTAL P.24

ANALYTICAL DETAILS

CONTROL No.

ROLL

START

END

(IN)

Project No. 2006

Residual Acrylamide

UV -409

V50 -53.75

VAO<sub>44</sub> -45

Quanticare B -37

Quanticare A -44.5

Liquid Azo -7.75

Microwave -4.25

Particle Size -8.25

9 0.875

10 100.75

} Blocking effects

The above results show that UV treatment gives on average a reduction of 409 ppm reduction in residual Acm. Excepting Liquid azo the thermals gave similar reductions of about 40-50 ppm. Particle size, microwaving and no. 9 interactions had little effect. Number 10 may be an interaction between other conditions.

A similar analysis was carried out on the viscosity result

V50 0

VAO<sub>44</sub> 0

QB -3.45

QA -5.125

Liquid Azo -0.125

UV -15.625

Microwave -0.375

Particle Size 1.125

9 -2.25

10 +5

I declare that this work was done by me

*afmer*

I declare that this work was explained to me and that I have understood it

## ARCHIVE DETAILS

CONTROL No.

ROLL

START

END

IND?

Project No. 2006

Bondar FEB 3

FPR No	Intensity	Duration	Stage	4	5
142-239	-	-	-	-	+
240	+	-	-	+	-
241	-	+	-	+	-
242	+	+	-	-	+
243	-	-	+	+	+
244	+	-	+	-	-
245	-	+	+	-	-
246	+	+	+	+	+

Intensity - of 366nm peak+ max. =  $2 \times 10^{-4}$   $\mu\text{W}/\text{cm}^2/\text{nm}$ - min. =  $4 \times 10^{-5}$   $\mu\text{W}/\text{cm}^2/\text{nm}$ Duration- 15 mins ( $\approx 80\%$  D/W)

+ 40 mins

Stage of Drying+ 1<sup>st</sup> 40 mins- 2<sup>nd</sup> 40 mins

I declare that this work was done by me

of her

I declare that this work was explained to me and that I have understood it





Project No. 2006

Bonza I2959 Optimization FED

Having done some initial investigation work using the photoinitiator Irgacure 2959\* (shown below) and found it to be successful



in reducing free acrylamide from 2900ppm to 30ppm, ~~then~~ it was decided to design an optimization FED. This FED was designed to investigate the effects of I2959 dose, <sup>current</sup>uv light dose, time/d. of UV treatment and mass of gel chip in the glass head.

The following shows the experimental design.

	I2959 (ppm)	Intensity	Time (mins)	Mass (g)	5	6	FPR No.
1	100	1	10	200	-	-	142 - 353
3	500	1	10	200	+	-	354
4	100	5	10	200	+	+	355
2	500	5	10	200	-	+	356
2	100	1	10	400	-	+	357
4	500	1	10	400	+	+	358
3	100	5	10	400	+	-	359
1	500	5	10	400	-	-	360
4	100	1	50	200	+	+	361
2	500	1	50	200	-	+	362
1	100	5	50	200	-	-	363
3	500	5	50	200	+	-	364
3	100	1	50	400	+	-	365
1	500	1	50	400	-	-	366
2	100	5	50	400	-	+	367
4	500	5	50	400	+	+	368

I declare that this work was done by me

*afmer*

I declare that this work was explained to me and that I have understood it

date

TOTAL P.39